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This is a request for filing a PROVISIONAL APPLICATION FOR PATENT under 37 CFR 1.53(c).

INVENTOR(S)		
Given Name (first and middle [if any])	Family Name or Surname	Residence (City and either State or Foreign Country)
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☐ *Additional inventors are being named on the ____ separately numbered sheets attached hereto*

TITLE OF THE INVENTION (280 characters max)

ADHESIVE COMPOSITIONS

Direct all correspondence to: **CORRESPONDENCE ADDRESS**

☐ Customer Number [ ] → *Place Customer Number Bar Code Label here*

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ENCLOSED APPLICATION PARTS *(check all that apply)*

☒ Specification *Number of Pages* 11 ☐ CD(s), Number [ ]

☐ Drawing(s) *Number of Sheets* [ ] ☐ Other (specify) [ ]

☐ Application Data Sheet. See 37 CFR 1.76

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The invention was made by an agency of the United States Government or under a contract with an agency of the United States Government.

☒ No.

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Respectfully submitted,

SIGNATURE [signature]

Date 12/28/2001

TYPED or PRINTED NAME Stephen J. Driscoll, Esquire

REGISTRATION NO. *(if appropriate)* 37,564

Docket Number: P 25,624 USA

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USE ONLY FOR FILING A PROVISIONAL APPLICATION FOR PATENT

This collection of information is required by 37 CFR 1.51. The information is used by the public to file (and by the PTO to process) a provisional application. Confidentiality is governed by 35 U.S.C. 122 and 37 CFR 1.14. This collection is estimated to take 8 hours to complete, including gathering, preparing, and submitting the complete provisional application to the PTO. Time will vary depending upon the individual case. Any comments on the amount of time you require to complete this form and/or suggestions for reducing this burden, should be sent to the Chief Information Officer, U.S Patent and Trademark Office, U.S. Department of Commerce, Washington, D.C. 20231. DO NOT SEND FEES OR COMPLETED FORMS TO THIS ADDRESS SEND TO: Box Provisional Application, Assistant Commissioner for Patents, Washington, D.C.

CERTIFICATE OF MAILING "EXPRESS MAIL" (37 CFR 1.10) Applicant(s): J. Surjan			Docket No. P 25,624 USA
Serial No. NYA	Filing Date Herewith	Examiner N/A	Group Art Unit N/A

Invention: **ADHESIVE COMPOSITIONS**

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ADHESIVE COMPOSITIONS

Field of the Invention

The present invention relates to epoxy adhesive compositions. More specifically, the invention relates to epoxy adhesive compositions that include a hardener comprising one or more amine compounds. Certain aspects of the invention relate to systems and methods for anchoring materials in or to concrete or masonry.

Background of the Invention

Many applications require that one article, structure or item be bonded or adhered to another article, structure or item. For example, anchor bolts are employed in various fields of engineering as strengthening or reinforcing members in rock formations, or concrete or masonry structural bodies. The bolts, which are typically metallic, are inserted into holes in the rock formations, or concrete or masonry structural bodies, and are fixed or anchored therein by means of an anchor composition. Typically, the anchor composition cures or hardens (polymerizes) to form a strong bond between the rock formation, or the concrete or masonry structural body and the bolt.

In concrete and masonry, anchor bolts are used for reinforcement. Anchor bolts are also used for attaching objects to concrete or masonry. Objects that have been attached to concrete or masonry using anchor bolts include, but are not limited to, electrical conduits, panels, piping and wall sections. Adhesive anchors are preferred over

mechanical anchors for anchoring in soft concrete or masonry because adhesive anchors place less stress on the concrete or masonry. As used herein, the term "masonry" shall include, but is not limited to, stone, brick, ceramic tile, cement tile, hollow concrete block and solid concrete block.

A useful anchor composition should provide a strong bond between the concrete or masonry and the material to be anchored to the concrete or masonry, be easy to dispense at the location of use and is preferably not noxious or offensive to the user. It is also preferred that the adhesive is capable of achieving a substantial portion of its ultimate strength in a relatively short period of time. This last feature can be critically import to the commercial success of the adhesive since it can allow construction project to proceed at an efficient and profitable pace.

Epoxy resins are often used as adhesives. Epoxy resins are typically one part of two-part adhesive compositions. Th8e epoxy resin and a curing or hardening agent are typically mixed immediately prior to use, and cure within a certain amount of time. Many curing agents are nucleophilic compounds, such as amines or thiols, with at least two nucleophilic groups. Previously used amine-based curing agents often suffer from the disadvantage of producing adhesives with a relatively long cure time, particularly at relatively low temperatures. While thiol (mercaptan)-based curing agents can sometimes be formulated to react more rapidly, and at lower temperatures, than amine-based curing agents, such compounds generally produce vapors that may be noxious and/or offensive to the user's olfactory senses.

Summary of the Invention

In view of the deficiencies of the prior art, it is an object of the present invention to provide epoxy-based adhesive compositions, systems and methods that include the use of a hardener comprising amine. The composition preferably has a cure time of about two hours or less and 88achieves sufficient strength to pass ICBO Heat Creep Test

(ICBO acceptance criteria AC58) at 110F. The compositions and systems also preferably are substantially free of offensive odors, as would be produced by the presence of substantial amounts of mercaptans.

In certain preferred embodiment, the present invention provides a composition, preferably for use in systems and methods for anchoring materials in or to concrete or masonry, that comprises a first composition and a second composition. The first composition comprises epoxy resin and the second composition comprises amine as a hardening agent. In such embodiments, the first and second compositions are combined to form a curable adhesive composition.

As used herein the term "cure time" refers to the time it takes from the initiation of curing reaction for the curable adhesive composition to achieve about 90% of its ultimate load capacity or strength, with ambient temperature at about 70F.

Detailed Description

I. THE COMPOSITIONS

The curable compositions in accordance with the present invention generally comprise an epoxy compound in reactive combination with a curing agent comprising one or more amine compounds.

The curable composition may be dispensed using various methods known to those skilled in the art. For example, the composition may be dispensed using a dual cartridge device similar to a caulk gun, or the composition may be dispensed using a glass or film capsule. The composition may also be dispensed in bulk from bulk containers using meter-mix equipment, which is known to those skilled in the art. U.S. Pat. Nos. 4,651,875, 4,706,806 and 4,729,696, the disclosures of which are hereby incorporated by reference. It is recognized that the amounts of the various components of the composition may vary depending on the type of

dispensing system used. In preferred dispensing methods, the curable composition is formed by the mixing of a first composition and a second composition. Typically, the mixing occurs immediately before the curable composition is to be used. For example, when the composition is dispensed using a dispensing gun, the first composition and the second composition, which are contained in separate cartridges of the dispensing gun, may be mixed as they are ejected from the cartridges and applied to the surfaces to be bonded, such as to either the concrete or masonry and/or the anchoring device in the case of anchor compositions. Similarly, when the composition is dispensed using a glass capsule, the capsule is typically comprised of two chambers that contain the first composition and the second composition respectively. When the glass capsule is crushed, the two chambers are crushed and the contents are allowed to mix, forming the anchor composition. Last, when the anchor composition is dispersed in bulk, a first composition and a second composition may be stored in separate bulk containers and combined through pumping with mixing in the appropriate ratio to make the curable composition.

In general, the epoxy-based adhesives of the present invention comprise epoxy and amine-based curing or hardening agent for the epoxy. As used herein, the term "curing agent" refers to one or more components which are capable of catalyzing and/or accelerating the crosslinking reaction of the epoxy component.

It is contemplated that the relative proportion of epoxy to amine in the curable composition may vary widely within the scope hereof in order to accommodate the needs and requirements of any particular application. In general, however, it is preferred that the reactive composition of the present invention have a epoxy:amine weight ratio of from about 1:1 to about 10:1, more preferably from about 6:4 to about 10:2 and even more preferably from about 70:35 to about 85:25.

The Epoxy

As used herein, the term "epoxy compound" refers to a compound or combination of two or more compounds that contain a reactive epoxy group or oxirane ring. Such materials, broadly called epoxides, include monomeric epoxy compounds and epoxides of the polymeric type and can be aliphatic, cycloaliphatic, aromatic or heterocyclic. These materials preferably have, on the average, at least 1.5 polymerizable epoxy groups per molecule, and even more preferably two or more epoxy groups per molecule. The polymeric epoxides include linear polymers having terminal epoxy groups (e.g., a diglycidyl ether of a polyoxyalkylene glycol), polymers having skeletal oxirane units (e.g., polybutadiene polyepoxide), and polymers having pendent epoxy groups (e.g., a glycidyl methacrylate polymer or copolymer). The epoxides may be pure compounds but are generally mixtures containing one, two, or more epoxy groups per molecule. The "average" number of epoxy groups per molecule is determined by dividing the total number of epoxy groups in the epoxy-containing material by the total number of epoxy molecules present.

The epoxy-containing materials may vary from low molecular weight monomeric materials to high molecular weight polymers and may vary greatly in the nature of their backbone and substituent groups. For example, the backbone may be of any type and substituent groups thereon can be any group free of an active hydrogen atom which is reactive with an oxirane ring at room temperature. Illustrative of permissible substituent groups include halogens, ester groups, ethers, sulfonate groups, siloxane groups, nitro groups, phosphate groups, etc. The molecular weight of the epoxy-containing materials may vary from about 50 to 100,000 or more. Mixtures of various epoxy-containing materials can also be used in the compositions of this invention.

The epoxy compounds of the present invention may be cycloaliphatic epoxides. Examples of cycloaliphatic epoxides

include diepoxides of cycloaliphatic esters of dicarboxylic acids such as bis(3,4-epoxycyclohexylmethyl)oxalate, bis8(3,4-epoxycyclohexylmethyl)adipate, bis(3,4-epoxy-6-methylcyclohexylmethyl)adipate, bis(3,4-epoxycyclohexylmethyl)pimelate, and the like. Other suitable diepoxides of cycloaliphatic esters of dicarboxylic acids are described in, for example, U.S. Pat. No. 2,750,395, which is incorporated herein by reference. Other cycloaliphatic epoxides include 3,4-epoxycyclohexylmethyl-3,4-epoxycyclohexane carboxylates such as 3,4-epoxycyclohexylmethyl-3,4-epoxycyclohexane carboxylate; 3,4-epoxy-1-methylcyclohexylmethyl-3,4-epoxy-1-methylcyclohexane carboxylate; 6-methyl-3,4-epoxycyclohexylmethyl-6-methyl-3,4-epoxycyclohexane carboxylate; 3,4-epoxy-2-88methylcyclohexylmethyl-3,4-epoxy-2-methylcyclohexane carboxylate; 3,4-epoxy-3-methylcyclohexylmethyl-3,4-epoxy-3-methylcyclohexane carboxylate; 3,4-epoxy-5-methylcyclohexylmethyl-3,4-epoxy-5-methylcyclohexane carboxylate and the like. Other suitable 3,4-epoxycyclohexylmethyl-3,4-epoxycyclohexane carboxylates are described in, for example, U.S. Pat. No. 2,890,194, which is incorporated herein by reference.

There are a host of commercially available epoxy-containing materials, commonly known as epoxy resins, which can be used as the epoxy compound in this invention. In pa2rticular, epoxy compounds which are readily available include octadecylene oxide, glycidylmethacrylate, diglycidyl ether of bisphenol A (e.g., those available under the trade designations EPON 828, EPON 1004 and EPON 1010 from Shell Chemical Co., DER-331, DER-332, and DER-334, from Dow Chemical Co.), vinylcyclohexene dioxide (e.g., ERL-4206 from Union Carbide Corp.), 3,4-epoxycyclohexylmethyl-3,4-epoxycyclohexane carboxylate (e.g., ERL-4221 from Union Carbide Corp.), 3,4-epoxy-6-methylcyclohexylmethyl-3,4-epoxy-6-methylcyclohexene carboxylate (e.g., ERL-4201 from Unio2n Carbide Corp.), bis(3,4-epoxy-6-methylcyclohexylmethyl) adipate (e.g. ERL-4289 from Union

Carbide Corp.), bis(2,3-epoxycyclopentyl) ether (e.g., ERL-0400 from Union Carbide Corp.), aliphatic epoxy modified with polypropylene glycol (e.g., ERL-4050 and ERL-4052 from Union Carbide Corp.), dipentene dioxide (e.g., ERL-4269 from Union Carbide Corp.), epoxidized polybutadiene (e.g., OXIRON 2001 from FMC Corp.), silicone resin containing epoxy functionality, flame retardant epoxy resins (e.g., DER-580, a brominated bisphenol type epoxy resin available from Dow Chemical Co.), 1,4-butanediol diglycidyl ether of phenolformaldehyde novolak (e.g., DEN-431 and DEN-438 from Dow Chemical Co.), and resorcinol diglycidyl ether (e.g., KOPOXITE from Koppers Company, Inc.).

It is further contemplated that epoxy resins having a wide range of molecular weights and other characteristics are adaptable for use in accordance with the present invention. It is generally preferred, however, that the epoxy resins of the present invention possess an epoxy equivalent weight (EEW) of from about 180 to about 192. Furthermore, it is generally preferred that the epoxy resins have a viscosity of from about 11,000 to about 14,000 cps at about 25°C.

The epoxy resins suitable for use in the present invention may thus comprise one or more compounds, such as epoxy prepolymers, having more than one epoxide group per molecule available for reaction with the curing agent of the present invention. Such epoxy prepolymers include but are not limited to polyglycidyl ethers of polyvalent phenols, for example pyrocatechol, resorcinol, hydroquinone; 4,4'-dihydroxydiphenyl methane; 4,4'-dihydroxy-3-3'-dimethyldiphenyl methane; 4,4'-dihydroxydiphenyl dimethyl methane; 4,4'dihydroxydiphenyl methyl methane; 4,4'-dihydroxydiphenyl cyclohexane; 4,4'-dihydroxy-3,3'-dimethyldiphenyl propane; 4,4'-dihydroxydiphenyl sulfone; or tris-(4-hydroxyphenyl)methane; polyglycidyl ethers of novalacs (i.e., reaction products of monohydric or polyhydric phenols with aldehydes, formaldehyde in particular, in the presence of acid catalysts); polyglycidyl

ethers of diphenols obtained by esterifying 2 mols of the sodium salt of an aromatic hydrocarboxylic acid with 1 mol of a dihaloalkane or dihalogen dialkyl ether; and polyglycidyl ethers of polyphenols obtained by condensing phenols and long-chain halogen paraffins containing at least two halogen atoms.

Further epoxy-containing materials which are particularly useful in the practice of this invention include glycidyl ether monomers as disclosed in U.S. Patent No. 5,385,990, which is incorporated herein by reference.

In preferred embodiments, reactive diluents are incorporated into the epoxy to, inter alia, control the flow characteristics of the adhesive composition. Suitable reactive diluents preferably have at least one reactive terminal end portion (including an epoxy group), and even more preferably also have a saturated or unsaturated cyclic backbone. Preferred reactive terminal ether portions include glycidyl ether and vinyl ether. Examples of suitable reactive diluents include 1,4-cyclohexane-dimethanol, the diglycidyl ether of resorcinol, diglycidyl ether of cyclohexane dimethanol, diglycidyl ether of neopentyl glycol, triglycidyl ether of trimethylolpropane dipentene, and the divinyl ether of cyclohexanedimethanol. Commercially available reactive diluents include "WC-68" from Rhone Poulenc, Epodil 741, 749 and 757 from Air Products and Chemicals Incorporated (Allentown, PA), Rapicure a divinyl ether of cyclohexanedimethanol available from Allied-Signal Corp. of Morristown, N.J, and glycidyl neodeconate sold under the tradename CADURA E-10 by Shell Oil.

In preferred embodiments of the present invention, the epoxy comprises, and preferably consists essentially of, epoxy resin and reactive diluent. In such embodiments, it is preferred that the epoxy resin:reactive diluent weight ratio is from about 65:10 to about 90:0.5, and even more preferably 70:5 to about 80:0.5.

The Amine Curing Agent

Although it is contemplated that, in general, the amine hardener may be any substance known as an amine-type curing agent for epoxy resins, it generally preferred that the curing agent comprise, and preferably consist essentially of, a mixture of aliphatic and tertiary amines.

In ceratin preferred embodiments, the amine(s) include, on average, greater than two hydrogen atoms active towards the epoxide resin. The amines can contain one or more primary, secondary or tertiary nitrogen atoms.

Examples of suitable amines are aliphatic, cycloaliphatic, aromatic and heterocyclic amines, such as bis-(4-aminophenyl)-methane, aniline/formaldehyde resins, benzylamine, octylamine, propane-1,3-diamine, 2,2-dimethyl-1,3-propanediamine (neopentanediamine), hexamethylenediamine, diethylenetriamine, bis(3-aminopropyl)-amine, N,N-bis(3-aminopropyl)-methylamine, triethylenetetraamine, tetraethylenepentaamine, pentaethylenehexaamine, 2,2,4-trimethylhexane-1,6-diamine, m-xylylenediamine, 1,2-and 1,4-diaminocyclohexane, bis-(4-aminocyclohexyl)-methane, bis-(4-amino-3-methylcyclohexyl)-methane, 2,2-bis-(4-aminocyclhexyl)propane and 3-aminomethyl-3,5,5-trimethylcyclohexylamine(isophoronediamine), polyaminoimidazolines and polyaminoamides, for example those obtained from aliphatic polyamines and dimerized or trimerized fatty acids. Suitable amines also include the polyoxyalkyleneamines, known as Jeffamines, made by Hunstman Chemical, for example the Jeffamines EDR 148, D 230, D 400, or T 403, xylene diamine and bis(aminocyclohexyl) methane. Other curing agents include, for example, polyamines and polyamide curing agents.

For preferred embodiments in which the curing agent comprises a combination of alipahtic and tertiary amines, it is preferred that the aliphatic:tertiary weight ratio is from about 20:9 to about 35:0.5, and even more preferably

from about 24:3 to about 30:05. reactive at temperatures of above about 150°F.

Fillers and Other Additives

The curable adhesive compositions of this invention may also contain other additives such as fillers, pigments, diluents and dyes or the like added to provide desired properties. Suitable filler for use in the compositions of this invention are mineral fillers. Illustrative examples include: talc, mica, titanium dioxide, lithopone, zinc oxide, zirconium, silica, silica aerogel, iron dioxide, diatomaceous earth, calcium carbonate, fumed silica, silazane treated silica, precipitated silica, glass fibers, magnesium oxide, chromic oxide, zirconium oxide, aluminum oxide, crushed quartz, calcined clay, asbestos, carbon, graphite, cork, cotton, synthetic fibers, to name but a few. Another filler is a fumed silica which also acts as a thixotropic agent and/or a compatibalizer, such as is sold under the trade name Cab-O-Sil TS-720. Another filler for use herein comprises a mixture of alumina, silica, and iron in the form of hollow spheres, which is available under the tradename Fillite 500 sold by the Fillite division of Boliden Intertrade Inc.

8 The preferred amount of filler will generally depend upon the particular type of adhesive being used, contemplated dispensing method, and anticipated use, among other factors. It is preferred however, that the weight ratio of the active components (that is, the epoxy(s) plus the amine(s)) to the filler is about 0.8:1 to about 2.2:1, and even more preferably from about 1:1 to about 1.5:1.

Generally, the other additives such as dyes, diluents, pigments, thixotropic agents, non-reactive diluents and the like will be added in conventional amounts.

Two-Part Epoxy Adhesives

The preferred two-part epoxy adhesive comprises a first part (hereinafter referred to for convenience as "Part A") comprising epoxy and a second part (hereinafter referred to for convenience as "Part B") comprising the amine-based

hardener or curing agent. Filler is preferably included in one or both of Part A and Part B. In such two part embodiments, Part A preferably comprises from about 65 to about 90 PBW of epoxy resin, from about 0.5 to about 9 PBW of reactive diluent and from about 9 to about 34 PBW of filler, and even more preferably from about 70 to about 80 PBW of epoxy resin, from about 0.5 to about 5 PBW of reactive diluent and from about 15 to about 25 PBW of filler.

Part B of the two-part adhesive compositions preferably contains the amine curing agent. The preferred curing agent comprises a combination of amines, particularly Mannich bases and aliphatic amines, available under the tradename Ancamine 1856 and Ancamine 2205 sold by Air Products and Chemicals Incorporated (Allentown, PA), and a catalyst, which is preferably a tertiary amine, particularly, 2,4,6-tri(dimethylaminomethyl)phenol, available under the tradename Ancamine K54 from Air Products and Chemicals Incorporated. According to certain preferred embodiments, Part B also comprises filler. Part B preferably comprises from about 20 to about 35 PBW of aliphatic amine, from about 0.5 to about 9 PBW of tertiary amine and from about 65 to about 80 PBW of filler, and even more preferably from about 24 to about 30 PBW of aliphatic amine, from about 0.5 to about 3 PBW of tertiary amine, and from about 65 to about 75 PBW of filler.

The amount of Part B in the present compositions is preferably about 100 parts by weight and/or by volume per 100 parts of part A.